

**PATENT COOPERATION TREATY**  
**PCT**  
**INTERNATIONAL PRELIMINARY EXAMINATION REPORT**  
(PCT Article 36 and Rule 70)

REC'D 14 FEB 2006

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REC'D 14 FEB 2006

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Applicant's or agent's file reference M80580405:BGC:DVL	<b>FOR FURTHER ACTION</b>	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/416).
International Application No. <b>PCT/AU2003/001336</b>	International Filing Date (day/month/year) 10 October 2003	Priority Date (day/month/year) 10 October 2003
International Patent Classification (IPC) or national classification and IPC  Int. Cl.  <b>C08K 3/20 (2006.01) C08K 3/22 (2006.01) C08K 3/34 (2006.01)</b> <b>C08K 3/14 (2006.01) C08K 3/30 (2006.01) C08K 3/36 (2006.01)</b>		
Applicant <b>COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al</b>		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 3 sheets, including this cover sheet.
- ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 5 sheet(s).

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 10 May 2005	Date of completion of the report 31 January 2006
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer  <b>ROSS OSBORNE</b> Telephone No. (02) 6283 2404

## Basis of the report

With regard to the elements of the international application:\*

☐ the international application as originally filed.☒ the description, pages 1-14, as originally filed,  
pages , filed with the demand,  
pages , received on with the letter of☒ the claims, pages , as originally filed,  
pages , as amended (together with any statement) under Article 19,  
pages , filed with the demand,  
pages 15-19, received on 10 January 2006 with the letter of 10 January 2006☒ the drawings, pages 1/5-5/5, as originally filed,  
pages , filed with the demand,  
pages , received on with the letter of☐ the sequence listing part of the description:

pages , as originally filed

pages , filed with the demand

pages , received on with the letter of

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).☐ the language of publication of the international application (under Rule 48.3(b)).☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.☐ filed together with the international application in computer readable form.☐ furnished subsequently to this Authority in written form.☐ furnished subsequently to this Authority in computer readable form.☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished4. ☐ The amendments have resulted in the cancellation of:☐ the description, pages☐ the claims, Nos.☐ the drawings, sheets/fig.5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).\*\*

\* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

\*\* Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims 1-49	YES
	Claims	NO
Inventive step (IS)	Claims 12-49	YES
	Claims 1-11	NO
Industrial applicability (IA)	Claims 1-49	YES
	Claims	NO

2. Citations and explanations (Rule 70.7)

D1 WO 2001057142

D2 EP 1125969

D3 EP 292120

D4 EP 345644

D5 EP 486225

D6 EP 506033

**New Citations**

D7 US 6203906 (Christie et al) 20 March 2001

D8 US 6046267 (Vidaurre et al) 4 April 2000

NOVELTY (N) Claims 1-49

D1-D7 disclose polymeric films and resins with inorganic filler particles in the size range and loadings claimed. The citations do not explicitly disclose a polymer article that is substantially free of gas bubbles or the methods of preventing gas bubbles claimed. Claims 1-49 are therefore novel.

INVENTIVE STEP (IS) Claims 1-11

Claims 1-11 do not require the polymeric articles to be made free of gas by any particular method.

D1-D7 disclose polymeric films and resins with inorganic filler particles in the size range and loadings claimed.

D8 discloses a method of degassing polymeric composites that are reinforced with particulate filler under reduced pressure conditions. To overcome problems with gas bubbles in reinforced polymeric product it would be obvious to use a reduced pressure degassing step in the preparation process as taught by D8. The use of particular filler particle types or size sub-ranges or restricting the polymer used to thermoplastic polymers are not considered pertinent to the question of inventive step. Claim 10 refers to the use of a hydrophobicity matching dispersion agent but this feature only appears to have significance in reducing bubble content when the particles are required to be milled under low shear conditions. For these reasons claims 1-11 lack an inventive step.

**CLAIMS**

1. A polymer article comprising a thermosetting polymer matrix and inorganic ultrafine particulate material evenly distributed through the polymer matrix characterised in that the inorganic particulate material has a particle size up to 10  
5  $\mu\text{m}$  at a particle loading of 0.01 – 20 wt% based on the total weight of the polymer inorganic components, wherein the polymer article is substantially free of gas bubbles.
2. The article of claim 1, wherein the particle size is in the range of 1 to 1000 nanometers.
- 10 3. The article according to claim 1, wherein the upper size limit of the particle is less than 800 nanometers.
4. The article according to claim 1, wherein the particulate material has a size range of between 100-800 nanometers.
5. The article according to any one of claims 1-4, wherein the upper particle  
15 loading limit is 10 wt% based on the total weight of polymeric and inorganic material.
6. The article of claim 1, wherein the interparticle distance between the particles is less than  $20\mu\text{m}$  and the average interparticle distance is in the range of  $2\mu\text{m}$  to  $10\mu\text{m}$ .
- 20 7. The article according to claim 1, wherein the inorganic particulate material is an inorganic material available in submicron powder form, or a precursor to said inorganic particulate material, selected from the group including metal oxides, metal silicates and metal alkoxides.
8. The article according to claim 7, wherein the particulate material is an  
25 oxide, silicate and alkoxide of aluminium, titanium and silicon.
9. The article according to claim 7, wherein the particulate material is selected from the group consisting of alumina, ( $\text{Al}_2\text{O}_3$ ), titania ( $\text{TiO}_2$ ), zirconia ( $\text{ZrO}_2$ ), silica ( $\text{SiO}_2$ ), silicon carbide, hydroxides of alumina and alumino- silicates.
10. The article according to claim 1, wherein the particulate material is coated  
30 or treated with a dispersing agent to match the hydrophobicity of the particles with

the polymer.

11. The article according to claim 1, wherein the polymeric material is a polyurethane, polyester, hybrid or copolymer thereof.

12. A method of forming a polymer-inorganic composite article comprising the  
5 steps of:

dispersing an inorganic submicron particulate material having a particle size up to 10  $\mu\text{m}$  in a polymer at a loading rate of 0.01 to 20 wt% of the total weight of polymer and inorganic material, the dispersion step preventing the formation of gas bubbles in the mixture to provide a dispersion having a substantially even  
10 distribution of particulate material in the polymer; and

casting the dispersion in a mould to cure the mixture.

13. The method of claim 12, wherein the particle size is in the range of 1 to 1000 nanometers.

14. The method according to claim 13, wherein the mixing is carried out to  
15 minimise the amount of agglomeration of dispersed particles and provide a substantially even distribution of particles in the polymer.

15. The method according to claim 14, wherein the mixing step includes milling the particulate material with the polymer under sub-atmospheric conditions.

16. The method of claim 15, wherein the mixing is carried out in a low shear  
20 regime having a Reynolds number less than or equal to 1000.

17. The method of claim 15, wherein the mixing is carried out in a high shear regime having a Reynolds number much greater than 1000.

18. The method of claim 16, wherein the particulate material is coated or treated with a dispersing agent to match the hydrophobicity of the particles with  
25 the polymer.

19. The method of claim 18, wherein the dispersing or compatibilizing agent is selected from the group consisting of non-alcohol or non amine aromatic solvent.

20. The method of any one of claims 12 to 19, wherein the mixing occurs at under vacuum conditions to ensure no bubbles are formed in the dispersion.

21. The method of any one of claims 12 to 19, wherein the mixing occurs under a pressure of less than one atmosphere to ensure no bubbles are formed in the dispersion
22. The method of claim 20 or 21, wherein the mixing is conducted in a vacuum oven at a temperature above melting temperature and below the decomposition temperature of the polymer.
23. The method of any one of claims 12 to 22, wherein the mixing takes place in a vacuum up to 760 mm Hg with the level dependent on the viscosity of the dispersion.
24. The method of claim 16, wherein the particulate material is mixed with a solvent prior to mixing with the polymer material, with the particulate material, the polymer material and the solvent forming a slurry.
25. The method of claim 24, wherein the solvent is removed by evaporation from the dispersion and the dispersion is cured to form a composite substantially free of gas bubbles.
26. The method of claim 17, wherein the inorganic particulate material is dispersed in the polymer by milling the particulate material and polymer at a mill temperature, above the melting temperature resin but below the decomposition temperature of the polymer resin at a pressure below atmospheric pressure.
27. The method of any one of claims 12 to 26, wherein after the mixing step polymerisation of the polymer is initiated by the addition of an initiator and the dispersion cured.
28. The method of any one of claims 12 to 26, wherein at least one other additive selected from the group consisting of anti static agents, fillers, pigments, optical brighteners and UV brighteners are added to the dispersion.
29. A method of dispersing an inorganic particulate material substantially homogenously in a polymer, the particulate material consisting essentially of particles having a particle size up to 10  $\mu\text{m}$  at a loading rate in the range of 0.01 to 20 wt% based on the total weight of the particulate and polymer material, the method comprising the steps of combining the particulate material and a powder of the polymer under sub atmospheric pressure conditions, the combination being

milled at a temperature above the melting temperature but below the decomposition temperature of the polymer to form a dispersion, and curing the dispersion.

30. The method of claim 29, wherein the particle size is in the range of 1 to  
5 1000 nanometers.

31. The method of claim 30, wherein the particulate material is added to a solvent carrier prior to addition to the polymer to form a slurry, the solvent carrier being evaporated from the dispersion.

32. The method of claim 30 or 31, wherein the milling is conducted at a  
10 pressure of typically less than 900 millibar (gauge).

33. A method of increasing the wear resistance of a polymer comprising the steps of

dispersing an inorganic submicron particulate material having a particle size up to 10  $\mu\text{m}$  in a polymer at a loading rate of 0.01 to 20 wt% of the total weight of  
15 polymer and inorganic material, the dispersion step preventing the formation of gas bubbles in the mixture to provide a dispersion having a substantially even distribution of particulate material in the polymer; and

casting the dispersion in a mould to cure the polymer

34. The method of claim 33, wherein the particle size is in the range of 1 to  
20 1000 nanometers.

35. The method according to claim 34, wherein the dispersing step is carried out to minimise the amount of agglomeration of dispersed particles and provide a substantially even distribution of particles in the polymer.

36. The method according to claim 35, wherein the mixing step includes milling  
25 the particulate material with the polymer under vacuum conditions.

37. The method of claim 36, wherein the mixing is carried out in a low shear regime having a Reynolds number less than or equal to 1000.

38. The method of claim 36, wherein the mixing is carried out in a high shear regime having a Reynolds number much greater than 1000.

39. The method of claim 37, wherein the particulate material is coated or treated with a dispersing agent to match the hydrophobicity of the particles with the polymer.
40. The method of claim 39, wherein the dispersing agent is PGMA.
- 5 41. The method of any one of claims 33 to 40, wherein the mixing occurs at under vacuum conditions to ensure no bubbles are formed in the dispersion.
42. The method of any one of claims 33 to 40, wherein the mixing occurs under a pressure of less than one atmosphere to ensure no bubbles are formed in the dispersion
- 10 43. The method of claim 41 or 42, wherein the mixing is conducted in a vacuum oven at a temperature above melting temperature and below the decomposition temperature of the polymer.
44. The method of any one of claims 33 to 43, wherein the mixing takes place in a vacuum up to 760 mm Hg with the level dependent on the viscosity of the
- 15 dispersion.
45. The method of claim 37, wherein the particulate material is mixed with a solvent prior to mixing with the polymer material, with the particulate material, the polymer material and the solvent forming a slurry.
46. The method of claim 45 wherein the solvent is evaporated from the
- 20 dispersion and the dispersion is cured to form a composite substantially free of gas bubbles.
47. The method of claim 38, wherein the inorganic particulate material is dispersed in the polymer by milling the particulate material and polymer at a mill temperature, above the melting temperature but below the decomposition
- 25 temperature of the polymer resin at a pressure below atmospheric pressure.
48. The method of any one of claims 33 to 47, wherein after the mixing step polymerisation of the polymer is initiated by the addition of an initiator and the dispersion cured.
49. The method of any one of claims 33 to 47, wherein at least one other
- 30 additive selected from the group comprising anti static agents, fillers, pigments, optical brighteners and UV brighteners are added to the dispersion.